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IS 63 (2006): Whiting for paint and putty [CHD 20: Paints, Varnishes and Related Products]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
पेन्ट और पुट्टी की वाइटिंग — विशिष्टि
(तीसरा पुनरीक्षण)

Indian Standard
WHITING FOR PAINT AND PUTTY —
SPECIFICATION
(*Third Revision*)

ICS 87.060.10

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NEW DELHI 110002

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

Whiting, a naturally occurring form of calcium carbonate, is used as an extender in paints and also in the manufacture of putty.

This standard was first published in 1950 and subsequently revised in 1964 and 1978. In the second revision, IS 2468 : 1963 'Whiting for putty' was amalgamated and the requirements of the standards was covered in this standard with additional requirements to meet the need of the paint industry.

In the second revision, two types of the material had been covered, namely, whiting for paint and whiting for putty. For whiting used as extender in paint industry three grades had been prescribed. Additional requirements for pH value, silica content, settling tendency and microscopic examination for whiting for paint and pH value for whiting for putty had also been included.

With the advancement of technology, there is change of the requirements in the paint industries. Microfine extenders are also in use. Since crystalline grade of whiting is serviced as calcite and amorphous grade of whiting is also suitable for making putty, only two grades of whiting, that is, Grade 1 and Grade 2 have been specified in this standard.

In this standard, additional requirements for matter soluble in water, water demand, colour, opacity and particle size distribution have also been included. Requirements for pH value, matter insoluble in hydrochloric acid, alumina and iron oxide have been modified.

The requirements of calcium carbonate are covered in IS 8767 : 1978 'Calcium carbonate, precipitated and activated for paints'.

This standard contains clause 6.1 which calls for agreement between the purchaser and the supplier.

The composition of the Committee responsible for the formulation of this standard is given in Annex K.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

WHITING FOR PAINT AND PUTTY — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for naturally occurring calcium carbonate, that is, whiting for manufacture of paints and putty.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
33 : 1992	Inorganic pigments and extenders for paints — Methods of sampling and test (<i>third revision</i>)
75 : 1973	Specification for linseed oil, raw and refined (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
266 : 1993	Sulphuric acid (<i>third revision</i>)
323 : 1959	Specification for rectified spirit (<i>revised</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1303 : 1983	Glossary of terms relating to paints (<i>second revision</i>)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 33 and IS 1303 shall apply.

4 GRADES

The material shall be of two grades:

- a) *Grade 1* — Amorphous, suitable for making putty and paints; and
- b) *Grade 2* — Crystalline, suitable for making paints.

5 REQUIREMENTS

5.1 Composition

The material shall contain 90 ± 5 percent by mass of

calcium carbonate (as CaCO_3) when tested as prescribed in A-1. The material shall not contain any free lime (CaO) when tested as prescribed in A-2.

5.2 Form and Condition

The material shall be in the form of a dry powder free from grit or in such a condition that it can be reduced to the powder form by crushing, without grinding action, under a palette knife.

5.3 Putty Making Property

The material of Grade 1, when kneaded with linseed oil, raw (IS 75) in suitable proportion shall form a coherent paste which after thorough working in the hands shall have good plastic quality without sliminess or stickiness that would render it difficult to handle and apply. In addition, it shall work readily and smoothly without crumbling or cracking, and after being moulded in place it shall hold its shape until set.

5.4 The material shall also comply with the requirements given in Table 1.

6 PACKING AND MARKING

6.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the supplier.

6.2 Marking

6.2.1 Each package shall be marked with the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer or his recognized trade-mark, if any;
- c) Net mass of the material;
- d) Batch No. or Lot No. in code or otherwise; and
- e) Month and year of manufacture.

6.2.2 BIS Certification Marking

Each package may also be marked with the Standard Mark.

6.2.2.1 The use of the Standard Mark is governed

Table 1 Requirements for Whiting for Paint and Putty
(Clauses 5.4 and 8.1)

Sl No.	Characteristic		Requirements for Grades		Methods of Test, Ref to	
			Grade 1	Grade 2	Annex of this Standard	Clause No. of IS 33
(1)	(2)		(3)	(4)	(5)	(6)
i)	Volatile matter, percent by mass, <i>Max</i>		0.50		—	8
ii)	Residue on sieve, percent by mass, <i>Max</i>		0.5 on 63 micron IS Sieve	0.2 on 37 micron IS Sieve	—	9
iii)	Oil absorption ¹⁾		13 to 22	15 to 25	—	10
iv)	Particle shape		Similar to approved sample		—	18
v)	Matter soluble in water, percent by mass, <i>Max</i>		1.00		—	19
vi)	pH of the aqueous extract		8 ± 1		—	21
vii)	Silica (as SiO ₂), percent by mass, <i>Max</i>		0.1		B	—
viii)	Settling tendency		To pass the test		C	—
ix)	Matter insoluble in hydrochloric acid, percent by mass, <i>Max</i>		0.50	—	D	—
x)	Alumina and iron oxide, percent by mass, <i>Max</i>		0.50	—	E	—
xi)	Water demand	Stiff point	17 ± 3	20 ± 5	F	—
		Flow point	19 ± 3	25 ± 5		
xii)	Colour		Close match to approved sample		G	—
xiii)	Whiteness and opacity		Close match to approved sample		H	—
xiv)	Particle size distribution, percent by mass, finer than 10 microns, <i>Min</i>		—	96	J	—

¹⁾ This shall, however, be within ±10 percent of the approved sample, if any.

by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

7.1 Preparation of Test Samples

Representative samples of the material shall be prepared as prescribed in 5 of IS 33.

7.2 Number of Tests

Tests for the determination of all the characteristics specified shall be conducted on the composite sample.

7.3 Criteria for Conformity

The material shall be taken as conforming to this standard, if the composite sample satisfies all the requirements prescribed in 5.

8 TEST METHODS

8.1 Tests shall be conducted as prescribed in Annexes A to J of this standard and in IS 33. Reference to relevant Annex and clauses of IS 33 appears in col 5 and 6 of Table 1.

8.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(Clauses 5.1 and 8.1)

ANALYSIS OF WHITING

A-1 DETERMINATION OF CALCIUM CARBONATE CONTENT

A-1.1 General

The calcium carbonate content is determined volumetrically, using standard potassium permanganate solution.

A-1.2 Reagents

A-1.2.1 Standard Potassium Permanganate Solution (0.1 N) — Dissolve 3.2 g of potassium permanganate in 1 000 ml of water and allow it to stand for 8 to 14 days. Siphon off the clear supernatant solution into dark-coloured glass-stoppered bottle. Weigh accurately about 3.3 g of sodium oxalate, previously dried for a few hours at $100 \pm 2^\circ\text{C}$ and cooled over fused calcium chloride in a desiccator. Dissolve it in water and make up the solution to exactly 500 ml. Transfer 25 ml of sodium oxalate solution to a conical flask, add 50 ml of water and 10 ml of dilute sulphuric acid (1 : 1 by volume). Heat the solution to about 60°C and titrate with potassium permanganate solution.

$$\text{Strength of permanganate solution} = 0.373 \times \frac{M}{V}$$

where

M = mass of sodium oxalate in 1 000 ml of solution, in g; and

V = volume of potassium permanganate required

for titration against 25 ml of sodium oxalate solution, in ml.

A-1.2.2 Bromine Water

A-1.2.3 Ammonium Oxalate Solution — Saturated.

A-1.2.4 Dilute Hydrochloric Acid — Add one volume of concentrated hydrochloric acid (*see* IS 265) to one volume of water.

A-1.2.5 Dilute Sulphuric Acid — Carefully add one volume of concentrated standard sulphuric acid (*see* IS 266) to 4 volumes of water.

A-1.3 Procedure

Transfer about 0.2 g of accurately weighed whiting, dried as described in 8 of IS 33 to a beaker. Dissolve the whiting in about 20 ml of dilute hydrochloric acid. Digest for 10 min on a steam bath, dilute to 150 ml, filter and wash the residue with water. Add a few millilitres of bromine water / nitric acid (2 to 3 drops) and heat to boiling and make the boiling solution ammoniacal. Filter off the residue, wash it thoroughly and reduce the filtrate by evaporation to 200 ml. To the slightly ammoniacal solution, heated to boiling, add an excess of hot ammonium oxalate solution. Continue boiling till the precipitate becomes granular. Allow to stand for 1h, filter and wash with hot water. Pierce the apex of the filter paper with a stirring rod and wash the precipitate into beaker with hot water. Pour warm dilute sulphuric acid through the paper and wash it a few times with warm acid. Add about 30 ml of dilute sulphuric acid, dilute to about

250 ml, heat to 60°C and titrate with standard potassium permanganate solution.

A-1.4 Calculation

$$\text{Calcium carbonate, percent by mass} = \frac{0.5004 V}{M}$$

where

V = volume of 0.1 N potassium permanganate solution used for titration, in ml; and

M = mass of the material taken for test, in g.

A-1.5 The carbon dioxide content shall also be estimated by a suitable method. This shall not be less than the equivalent value for calcium carbonate (CaCO_3) content.

A-2 TEST FOR FREE LIME

A-2.1 Reagents

A-2.1.1 Cane Sugar Solution — Dissolve 10 g of cane

sugar in 100 ml of water.

A-2.1.2 Phenolphthalein Indicator Solution — Dissolve 0.1 g in 100 ml of rectified spirit (see IS 323).

A-2.1.3 Standard Hydrochloric Acid — 0.1 N.

A-2.2 Procedure

Grind the material to a fine powder, take about 0.1 g of it in a flask, shake vigorously with about 30 ml of cane sugar solution, filter and test the filtrate with phenolphthalein indicator. If pink colour is developed, add to the solution 3 drops of standard hydrochloric acid.

A-2.2.1 The material shall be deemed to contain no free lime, if on the addition of the indicator no colour is developed; or if any pink colour is developed, it is discharged by the addition of 3 drops of standard hydrochloric acid.

ANNEX B

[Table 1, Sl No. (vii) and Clause 8.1]

DETERMINATION OF SILICA

B-1 REAGENTS

B-1.1 Sulphuric Acid (1:1) — Add 1 volume of sulphuric acid, relative density 1.84, slowly to 1 volume of water.

B-1.2 Hydrofluoric Acid — 40 percent (m/m).

B-2 APPARATUS

B-2.1 Platinum Dish

B-2.2 Muffle Furnace

B-3 PROCEDURE

B-3.1 Weigh accurately about 1 g of the sample dried as prescribed in 8 of IS 33, in the platinum dish and add about 1 ml of the sulphuric acid. Heat the dish gently until fuming ceases and then continue heating at $900 \pm 25^\circ\text{C}$ for 15 min in the muffle furnace. Remove from the furnace, cool in a desiccator and weigh.

B-3.2 Add to the residue in the platinum dish 15 ml

of the hydrofluoric acid and 1 ml of the sulphuric acid and evaporate to a syrup, taking care to avoid loss by spurring. Cool the dish and wash the sides down with water. Then add a further 10 ml hydrofluoric acid and evaporate to dryness. Heat the residue on a hot plate until white fumes are no longer evolved, then ignite in the muffle furnace at $900 \pm 25^\circ\text{C}$ for 15 min.

B-3.3 Remove from the furnace, cool in a desiccator and weigh.

B-4 CALCULATION

$$\text{Silica content, percent by mass} = \frac{m_2 - m_3}{m_1} \times 100$$

where

m_2 = mass after igniting with sulphuric acid, in g;

m_3 = mass after igniting with hydrofluoric acid, in g; and

m_1 = mass of the sample, in g.

ANNEX C

[Table 1, Sl No. (viii) and Clause 8.1]

TEST FOR SETTLING TENDENCY**C-1 APPARATUS****C-1.1 Stoppered 100 ml Cylinder** — Graduated.**C-1.2 Water Bath of Temperature**, $25 \pm 1^\circ\text{C}$ **C-1.3 Thermometer for Water Bath****C-2 PROCEDURE**

Weigh accurately 20 g of the whiting on butter paper and transfer it to the clean 100 ml measuring cylinder. Add water to make volume 100 ml. Thoroughly shake the cylinder after putting glass stopper. In another measuring cylinder of similar dimensions take 20 g of approved sample and make volume, by adding water, to 100 ml. Shake thoroughly. Keep both the

cylinders in the water bath for 30 min, maintained at $25 \pm 1^\circ\text{C}$ temperature. Remove both measuring cylinders and shake thoroughly and again keep in the water bath. Note the time. Observe the cylinders after 1 h without disturbing. The sample shall have settled layer comparable to or less than that of approved sample. Also observe the top layer which shall have clarity comparable to that of approved sample. Not (if necessary) the reading of the cylinder, both for the bottom settled layer and top clear solution.

C-3 REPORT

The material shall be taken to have passed this test if the sample under test shall show a comparable behaviour to that of the approved sample.

ANNEX D

[Table 1, Sl No. (ix) and Clause 8.1]

DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID**D-1 REAGENT****D-1.1 Dilute Hydrochloric Acid** — approximately 5 N.**D-2 PROCEDURE**

Weigh accurately about 2 g of the material, previously dried at $105 \pm 2^\circ\text{C}$ to constant mass, into a 250-ml beaker, add 50 ml of dilute hydrochloric acid. Boil for 10 min, dilute to 100 ml and filter through a filter paper (Whatman No. 40 or its equivalent). Wash the filter paper with hot water until the filtrate is free

from chlorides. Dry the filter paper and transfer it into a weighed crucible previously ignited. Heat the crucible on a burner and subsequently in a muffle furnace at $900 \pm 25^\circ\text{C}$. Weigh the crucible and contents after cooling. Repeat heating, cooling and weighing until the mass is constant.

D-3 RESULTS

Express the mass of the ignited residue as a percentage of the mass of the dried material taken for the test.

ANNEX E

[Table 1, Sl No. (x) and Clause 8.1]

DETERMINATION OF ALUMINIUM AND IRON OXIDE**E-1 REAGENTS****E-1.1 Ammonium Chloride****E-1.2 Ammonium Nitrate Solution** — Approximately 2 percent (m/v).**E-1.3 Ammonium Hydroxide Solution** — Relative density 0.90.**E-1.4 Dilute Hydrochloric Acid** — Approximately 25 percent (m/v).**E-1.5 Concentrated Nitric Acid** — Relative density 1.42.**E-2 PROCEDURE**

E-2.1 Weigh accurately about 2 g of the material dried as prescribed in 8 of IS 33 and transfer to a 250-ml beaker. Add 50 ml of dilute hydrochloric acid, boil for 10 min and dilute to 100 ml. Filter through a filter paper (Whatman No. 40 or its equivalent). Wash the filter paper with hot water until the filtrate is free from chlorides. Collect the filtrate

and washings in a beaker, add 2 g of the ammonium chloride and then a few drops of concentrated nitric acid. Boil for 5 to 10 min. Render the contents slightly alkaline with ammonium hydroxide solution and bring to boil. Allow the beaker to stand in a warm place for half an hour and filter through a filter paper (Whatman No. 40 or its equivalent). Replace the beaker containing the filtrate by original beaker and dissolve the precipitate with a few millilitres of dilute hydrochloric acid followed by hot water. Add 2

g of ammonium chloride and precipitate with ammonium hydroxide solution. Boil and filter through a filter paper (Whatman No. 41 or equivalent). Wash the precipitate with ammonium nitrate solution, dry along with filter paper and ignite at $900 \pm 25^\circ\text{C}$.

E-2.2 Calculation

Express the mass of the residue as percentage of the mass of the dried material taken for the test.

ANNEX F

[Table 1, Sl No. (xi) and Clause 8.1]

DETERMINATION OF WATER DEMAND

F-1 REAGENTS

F-1.1 0.5 percent (w/v) sodium hexameta phosphate (A.R. grade) solution in water.

F-2 APPARATUS

F-2.1 Burette — 50 ml.

F-2.2 Beaker — 100 ml.

F-2.3 Glass Rod of Suitable Length for Stirring

F-2.4 Weighing Balance (With 0.01 g Precision)

F-3 PROCEDURE

F-3.1 Test for Stiff Point

Weigh 10 g of sample (nearest to second place of decimal) in a 100-ml beaker. Add sodium hexameta phosphate solution drop by drop carefully with constant stirring with the glass rod. Stiff point is indicated when there is no more free particles of powder, that is, when the particles are converted into lump. Note the reading on the burette and calculate stiff point (V_1).

F-3.2 Test for Flow Point

Continue addition of sodium hexameta phosphate solution, drop by drop in the beaker (see F-3.1) with simultaneous stirring of the paste form, till the paste obtained is having consistency such that after inclining the beaker by 45° , the paste just starts flowing. Note the reading on the burette and calculate the flow point (V_2).

F-4 CALCULATION

$$\text{Stiff point} = \frac{V_1 \times 100}{M}$$

$$\text{Flow point} = \frac{V_2 \times 100}{M}$$

where

V_1 = volume of solution required for converting the sample into paste, in ml;

V_2 = volume in ml of solution required for the sample to reach flow point condition, in ml; and

M = mass of the sample taken for test, in g.

ANNEX G

[Table 1, Sl No. (xii) and Clause 8.1]

DETERMINATION OF COLOUR

G-1 GENERAL

The colour of a coloured pigment is compared with that of an approved sample.

G-2 REAGENTS

G-2.1 Soyabean Oil

G-3 APPARATUS

G-3.1 Palette Knife — Tapered steel blade of approximately 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

G-3.2 Plate, Ground Glass or Marble

G-3.3 Glass Panel — Clear colourless glass of minimum area 150 mm × 50 mm.

G-4 PROCEDURE

G-4.1 Take approximately 5 g of sample. Put soyabean oil drop wise on the sample and make a thick paste to apply on the panel.

G-4.2 Repeat the same as in G-4.1 with the approved sample.

G-4.3 Apply both the pastes side by side on the glass panel. The strips shall be of approximately 25 mm wide with touching the edges not less than 40 mm long.

G-4.4 Compare the colour by examining the strips in diffuse day light on the surface through the glass immediately after application. Where good day light is not available, make the comparison in artificial day light.

ANNEX H

[Table 1, Sl No. (xiii) and Clause 8.1]

DETERMINATION OF WHITENESS AND OPACITY

H-1 REAGENTS

H-1.1 Soft Water

H-1.2 Anionic Wetting Agent

H-1.3 Non-ionic Wetting Agent

H-1.4 Hydroxy Ethyl Cellulose

H-1.5 Any emulsion resin used for emulsion paint.

H-2 APPARATUS

H-2.1 Container — 200 ml.

H-2.2 Mechanical Stirrer — Laboratory grade.

H-3 PROCEDURE

H-3.1 Mix the following ingredients with the help of the stirrer in the order as stated below to make a uniform gel:

Soft water : 20.00 P.b.w.

Anionic wetting agent : 0.50 P.b.w.

Non-ionic wetting agent : 0.50 P.b.w.

Hydroxy ethyl cellulose : 0.20 P.b.w.

H-3.2 Add 50 g of the material under test slowly with the gel under stirring to make a uniform and homogeneous paste.

H-3.3 Add 25 g emulsion resin to the mixture in slow speed of stirring and homogenize.

H-3.4 Add 4 ml soft water and mix for 5 min.

H-3.5 Repeat H-3.1 to H-3.4 with the approved sample.

H-3.6 Make draw down of the above two pastes side by side on a contrast ratio paper (black and white 50 : 50) with K7 Bar Coater (wet film thickness approximately 75 to 80 micron).

H-3.7 Dry at 45°C for 2 h.

H-3.8 Compare the opacity and whiteness visually.

ANNEX J

[Table 1, Sl No. (xiv) and Clause 8.1]

DETERMINATION OF PARTICLE SIZE DISTRIBUTION

J-1 GENERAL

Because of the ease of execution and good reproducibility the Andreassen method is included as the referee method. Other methods may, however, be used by agreement between the concerned parties, but in such cases it will be necessary also to agree on appropriate limits.

J-2 PRINCIPLE

The rate of fall of spherical particles through a medium in which they are dispersed is proportional to the square of the particle diameter (Stokes' law). The method for determination of particle size distribution makes use of this relationship and expressed the particle size distribution in terms of distribution of spherical particles of the same settlement rate. In this determination, a dilute suspension is prepared and the concentration of solids at a fixed point below the surface is determined at a series of time intervals calculated to correspond to certain equivalent spherical diameters.

J-3 APPARATUS — see Fig. 1.

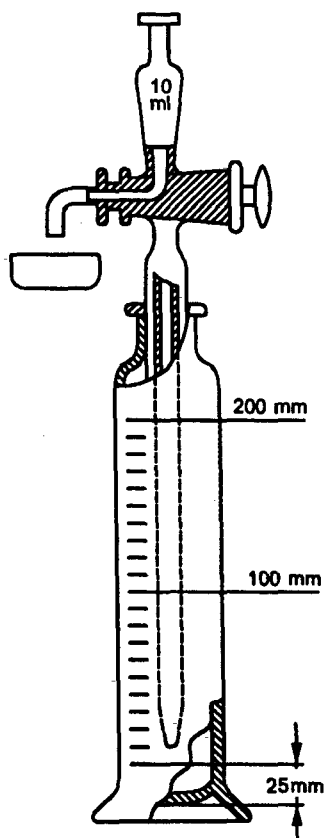


FIG. 1 SEDIMENTATION VESSEL AND PIPETTE

J-3.1 Sedimentation Vessel, of glass about 56 mm internal diameter and having a graduated scale from 0 to 200 mm marked on its side. The zero graduation line shall not be less than 25 mm from the inside base of the vessel and the capacity of the vessel up to the 200 mm mark shall be about 550 to 620 ml.

J-3.2 Pipette, fitted with a two-way tap and side discharge tube. The capacity of the pipette to the graduation line is conveniently 10 ml. A bell-shaped dome with a ground glass joint to fit the neck of the sedimentation vessel is fused to the pipette. A small vent hole is made in this dome. The inlet to the pipette stem shall be level with the zero line on the sedimentation vessel. The stem from the pipette bulb to the sampling inlet shall be constructed of capillary glass tubing with a bore not less than 1 mm and not more than 1.3 mm. The tube above the bulb shall be 4 to 4.5 mm bore.

J-3.3 Constant — Temperature Bath — Transparent-sided of at least 15 litres capacity, maintained at a temperature of $27 \pm 0.5^\circ\text{C}$, into which the sedimentation vessel can be immersed up to the 200 mm graduation mark. The bath shall be positioned away from sources of vibration.

J-3.4 Mechanical Stirrer — Capable of rotating at a suitable speed for complete dispersion ($1\,000 \pm 100$ rpm is generally suitable). The stirrer shall be designed to lift the dispersion and to avoid the creation of a vortex.

NOTE — A suitable stirrer may be made from an approximately 40 mm diameter brass disc with four equally spaced cuts, the cut sections being turned upwards at an angle of 30° to the horizontal.

J-3.5 Dispersion Vessel — Of appropriate dimensions, such as a 1 000 ml gas jar.

J-3.6 Balance — Having a sensitivity of not less than 0.1 mg.

J-3.7 Drying Oven — Capable of being maintained within a temperature range suitable for evaporation of the suspending liquid.

J-3.8 Weighing Bottles — Wide-mouthed, suitable for evaporation, of capacity not less than 20 ml, or a small laboratory centrifuge and centrifuge tubes preferably of 20-ml capacity but of not less than 10-ml capacity.

J-3.9 Stop-Watch or Stop-Clock

J-4 PREPARATION FOR THE TEST

J-4.1 Calibration of Pipette

Thoroughly clean the pipette. Partly fill the sedimentation vessel with water. Set the top in the sampling position and by means of a rubber tube suck water into the bulb to the level of the graduation line. Reverse the tap to the discharge position and allow the water to drain into a tared weighing bottle. Apply pressure through the rubber tube to blow any water remaining in the bulb and the discharge tube into the weighing bottle. Weigh the bottle to the nearest 0.001 g and calculate from this mass the internal volume V_p of the pipette.

J-4.2 Calibration of Sedimentation Vessel

Thoroughly clean the sedimentation vessel, weigh it, with the pipette in place, to the nearest 0.1 g. Then fill it to the 200 mm mark with water at $27 \pm 0.5^\circ\text{C}$ and reweigh. Calculate from the mass of water the internal volume V_g of the vessel.

J-4.3 Sample for Analysis

Using 3.5 g of accurately weighed material with 0.07 to 0.14 g of suitable dispersing materials (*see Note*), prepare a dispersion by placing the test portion in the dispersion vessel adding dispersion solution at $27 \pm 0.5^\circ\text{C}$ to give a total volume of about 500 ml and stirring with the mechanical stirrer for 15 min. Immediately pour all the suspension into the sedimentation vessel and make up to the 200 mm line with water or aqueous alcohol as appropriate. Transfer the sedimentation vessel to the constant temperature bath.

NOTE—Certain sodium salts of polymethacrylates have been found most suitable.

J-5 PROCEDURE

J-5.1 Sedimentation

Allow the sedimentation vessel to stand immersed to the 200 mm mark until it has reached the temperature of the bath. Record this temperature, which shall not differ greatly from that of the room. When temperature equilibrium has been attained, mix the contents thoroughly by placing a finger over the vent hole and inverting the vessel several times. Immediately after mixing, replace the vessel and start the stop-clock or stop-watch.

J-5.2 Withdraw from the suspension by means of the pipette a series of samples of volume V_p at depths h_n at increasing time intervals after mixing, starting at time t_1 corresponding to a particle size of about 20 micron (calculated in accordance with J-5.3) and thereafter in progression so that the limiting diameters stand in a $\sqrt{2}$:1 progression. Alternatively, the samples may be taken at convenient times approximately in a

2:1 progression and the exact spherical particle diameters corresponding to each sample calculated. The time to fill the pipette shall be about 20 s.

J-5.3 Discharge the suspension from the pipette into a tared weighing bottle. When the bulb has drained, remove the rubber suction tube and run 5 to 7 ml of water from an ordinary 10 ml pipette into the bulb of the sedimentation pipette to wash into the weighing bottle any particles adhering to the surface. The stem of the sedimentation pipette shall remain filled with suspension. Dry the contents of the weighing bottle at 105°C until the difference between successive weighings is not greater than 0.1 mg. Calculate the mass of the fraction m_n allowing for the mass of dispersing agent, which shall be determined by carrying out a blank test. The determination of different fractions may be carried out by means of chemical methods if that procedure is more suitable.

J-5.4 The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment and allow for it in the subsequent calculation. Suppose that the decrease is 4 mm for each fraction withdrawn, and that initially the depth of immersion of the pipette was 200 mm, then the depth after the first fraction has been withdrawn will be 196 mm, and the mean depth h_1 used for calculation of the initial diameter corresponding to the first fraction will be 198 mm. The mean depth h_2 for the second fraction will be 194 mm, and so on.

J-5.5 Calculation of Withdrawal Time

The withdrawal time, t_n , in seconds, for the n th sample is given by the equation:

$$t_n = \frac{18\sigma h_n}{(\mu_2 - \mu_1) g d_n^2} \times 10^6$$

where

σ = absolute viscosity of the medium, in NS/m²;

h_n = depth, at which the n th sample extraction is made, in mm;

μ_1 = density of the medium, in megagram/m³;

μ_2 = density of the particle, in megagram/m³ or g/cm³;

g = acceleration due to gravity, in m/s²; and

d_n = limiting stokes diameter corresponding to the n th sample extraction, in μm .

J-6 CALCULATION

J-6.1 The cumulative percentage by mass, P_n , of particles smaller than each of the limiting stokes diameter d_n for each time interval t_n is given by the formula;

$$P_n = \frac{m_n \times V_g}{m_g \times V_p} \times 100$$

where

m_n = mass of the fraction corrected for the mass of the dispersing agent, in g;

V_g = volume of the sedimentation vessel, in ml;

m_g = mass of the test portion, in g; and

V_p = volume of the pipette, in ml.

J-6.2 Repetition of Test

Repeat the procedure on a further amount of suspension

prepared from the same sample. The results of the test shall be accepted only if the percentages by mass, less than the same limiting stokes diameter, do not differ by more than 4 percent.

J-6.3 Expression of Results

Plot the results of the analysis with the micrometric sizes as abscissae and the percentages undersize as ordinates. From the smooth curve drawn through the points, select the cumulative percentages corresponding to the series required. Repeat the results to the nearest 1 percent.

ANNEX K

(Foreword)

COMMITTEE COMPOSITION

Paints, Varnishes and Related Products Sectional Committee, CHD 20

<i>Organization</i>	<i>Representative(s)</i>
In personal capacity (14, Orion, Oomer Park, Bhulabhai Desai Road, Mumbai)	SHRI RAVI MARPHATIA (<i>Chairman</i>)
Addisons Paint & Chemicals Ltd, Chennai	SHRI R. SRINIVASAN
Akzo Nobel Coatings India Pvt Ltd, Bangalore	SHRI DEEPAK VERMA
Asian Paints (India) Ltd, Mumbai	SHRI A. B. MENON DR B. P. MALIK (<i>Alternate</i>)
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Consumer Unity and Trust Society (CUTS), Jaipur	SHRI SANDEEP SINGH
Continental Coatings Pvt Ltd, Chennai	SHRI M. B. SATYANARAYANA
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Ministry of Environment & Forest, New Delhi	REPRESENTATIVE

<i>Organization</i>	<i>Representative(s)</i>
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BIS Directorate General	DR U. C. SRIVASTAVA, Scientist 'F' and Head (Chem) [Representing Director General (<i>Ex-officio</i>)]

Member Secretary
DR R. K. BAJAJ
Scientist 'E' (Chem), BIS

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$$\begin{cases} 2323 \ 7617 \\ 2323 \ 3841 \end{cases}$$
$$\begin{cases} 2337\ 8499, 2337\ 8561 \\ 2337\ 8626, 2337\ 9120 \end{cases}$$
$$\begin{cases} 260\,3843 \\ 260\,9285 \end{cases}$$
$$\begin{cases} 2254\ 1216, 2254\ 1442 \\ 2254\ 2519, 2254\ 2315 \end{cases}$$
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